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Abstract

A research program is under way to establish the feasibility of fabricating ion engine accelerator system electrodes from polycrystalline diamond film. Because of its high thermal conductivity, low coefficient of thermal expansion, and low erosion rate, diamond film has the potential of mitigating life limiting and performance degrading processes which have hindered flight qualification of ion thrusters to date. The relative erosion rates of polycrystalline diamond film, carbon-carbon, and molybdenum samples have been measured at incident ion energies of 500 and 750 eV with xenon. At 500 eV the relative erosion rates of molybdenum to carbon-carbon were found to be 7.3:1 and for carbon-carbon to diamond 1.6:1. At 750 eV the ratio was 7.7:1 for molybdenum to carbon-carbon and 1.9:1 for carbon-carbon to diamond. Three approaches are discussed for utilizing diamond's properties in the fabrication of thruster grids. In the first process a molybdenum grid is coated with a chemically vapor deposited diamond film. In the second approach, a thick (1 mm) diamond film is deposited on a substrate and then removed to produce a free-standing structure. The final approach involves the coating of a carbon-carbon composite grid. In each of these approaches, the diamond is doped to make it semiconducting.

1. Introduction and Background

Work conducted in recent years has significantly improved our understanding of the physical mechanisms which limit the lifetime of ion thrusters. Foremost among these mechanisms is the erosion of the accelerator grid (in

two grid optics) from bombardment with charge exchange (CEX) ions created in a region immediately downstream of the thruster¹.

As early as the 1960's, efforts to characterize the sputter yield of various elements had identified the improved resistance to erosion of carbon as compared with molybdenum^{2,3} when bombarded with xenon and krypton ions. As a result of its lower erosion rate, as well as its negative coefficient of thermal expansion, a program to develop and test ion optics fabricated from carbon fiber-carbon resin (carbon-carbon) was initiated at the Jet Propulsion Laboratory⁴.

More recently, advances in the chemical vapor deposition (CVD) of polycrystalline diamond films has prompted an effort to assess the potential benefits of diamond and the feasibility of applying some of these CVD processes to further improve upon the carbon based grid technology. This paper summarizes the status of this ongoing work.

Currently, two general strategies are under consideration to maximize the potential benefit of CVD diamond for thruster grids. Because the primary concern is erosion of the grid exposed to CEX ion impingement, the first strategy involves coating a molybdenum or carbon-carbon grid with a film of diamond. The second involves growing a diamond film grid thick enough (1mm) to be self supporting and capable of gentle handling. In either case the use of diamond would be limited to the accelerator grid (in two grid optics) or the decelerator grid (for three grid optics). For the purposes of this paper, discussion will be limited to a two grid system where the diamond is used only for the accelerator grid.

While many of the thermal, electrical, and mechanical properties of diamond have been well characterized, a review of the literature did not reveal sputter yield data for diamond under bombardment with xenon ions. As a result the measurement of these yields and the corresponding erosion rates has been the highest priority of this work to

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date. Of particular interest is the erosion rate of CVD diamond as compared to carbon-carbon and molybdenum.

The next section will briefly discuss the thermal and electrical properties of diamond as they pertain to the requirements of the accelerator grid for the ion thruster. This will be followed by a detailed description of the experiment used to evaluate the sputter yield and a summary of the data collected to date. The following section will briefly discuss the deposition process as well as some of the approaches under consideration for diamond grid fabrication. Finally the status of the evaluation and conclusions thus far will be presented.

II. Material Properties

A great deal of interest has surrounded the development of CVD diamond in recent years. Much of this interest is due to the unique combination of desirable properties found in both natural diamond as well as high quality polycrystalline diamond deposited from a vapor phase. Three of the properties most relevant to the application of diamond to ion optics are discussed below.

Thermal Conductivity

In a ion thruster operating using a hollow cathode electron source in the discharge chamber, the thermal environment to which the grids are subjected will be dominated by the cathode as well as the chamber or anode walls. Once the thruster is in steady state operation the cathode temperature will be close to 1200 C while the discharge chamber walls may be 400 C. While the screen grid will shield a fraction this radiant heat from the accelerator grid, the temperature could still reach a value of several hundred degrees Celsius unless the heat is removed. In addition the accelerator grid will be subject to heating from impingement of CEX ions on the downstream face. At these temperatures, the dominant heat transfer mechanism is conduction which one seeks to maximize in order to lessen the degree to which the grid will distort due to thermal expansion.

Table 1. Thermal Conductivity

Material	κ (W/m/K)	Notes
Molybdenum	138	20 c
P-100	520	21 c
Diamond	2000	20 c
Copper	401	20 c

Table 1 lists the room temperature thermal conductivities of molybdenum, P-100 (a commercial y available carbon-carbon composite), diamond and copper. Diamond has an unsurpassed thermal conductivity and the conductive cooling of the grid would be limited by the impedance of the grid-thruster mounting interface.

Coefficient of Thermal Expansion

As previously mentioned, heating of the accelerator grid results in a lateral deflection as a result of the longitudinal thermal expansion. This is a consequence of the fact the grid is rigidly fixed in the mounting ring along its perimeter. For a given steady state temperature during operation one would like to minimize this distortion. One reason is that the deflection of the grid surface will alter the spacing between the grids which is nominally on the order of 0.5 mm. Changes in the spacing will alter the attainable beam current for a given beam voltage and if severe enough could result in contact between the grids.

Table 2. Coefficient of Thermal Expansion

Material	α (10^{-6} /K)	Notes
Molybdenum	5.43	527 C
P-100	-1.5	527 C
Diamond	3.8	527 C

Table 2 two lists the CTE for molybdenum, P-100, and diamond. The P-100 composite is the most desirable in this respect because of its negative CTE. A negative CTE implies a contraction of the material upon heating which will tend to stiffen the grid making it more rigid. Diamond however is seen to have a CTE 30 percent lower than that of molybdenum.

Electrical Resistivity

Diamond has a band gap of 5.5 eV making it an excellent electrical insulator with a breakdown field of 10^9 V/m. Chemically vapor deposited diamond is routinely doped with boron to make it a p-type semiconductor. It is more difficult to make an n-type although this has also been attempted with mixed results by implantation of ions such as carbon or doping with phosphorus^{5,6}.

Ideally, the electrical current which must be conducted by the accelerator grid is only that due to CEX ion impingement and should not exceed several tens of milliamperes at a grid bias of several hundred volts. As the data in Table 3 illustrates, even with doping, the resistivity of diamond is significant. This should not be a

significant concern however as the currents involved are small (20-30 mA) and the effect of resistive heating will be mitigated by the high thermal conductivity and low CTE.

Table 3. Electrical Resistivity

Material	ρ ($\mu\Omega$ m)	Notes
Molybdenum	0.5	
P-100	2.5	
Diamond	$14.2 \cdot 10^6$	varies with doping

III. Sputter Yield

Background

Charge exchange ions are formed when a slow neutral atom collides with a fast ion resulting in a slow ion and a fast neutral. These are produced in a region extending a few millimeters downstream of the ion thruster. The source of the slow neutrals is a flux of un-ionized gas atoms which escapes the discharge chamber through the grids. In ground based tests, an additional source of neutrals will be the background gas in the test facility.

Several studies which have investigated this process and attempted to model it using particle-in-cell (PIC), and Monte-Carlo simulations⁷⁻⁹. The slow ions produced in the CEX reactions have insufficient kinetic energy to escape the negative potential well created by the accelerator grid and are accelerated back towards the thruster where they can impinge on the downstream face of the grid. Over time, this process will lead to the erosion of the webbing between holes in the grid resulting in structural weakening and increased neutralizer electron back streaming.

The potential to reduce this erosion was the primary motivation to incorporate diamond in the fabrication of the grids. However no published literature was found with sputter yield data for xenon on diamond. The energy range of interest corresponds to the kinetic energy of the CEX ions impinging on the grid which will depend on where they are formed as well as the bias of the accelerator grid. This value will be in the range of 250-1000 eV depending on the operating point chosen for a particular mission.

Experiment

The purpose of the experiment was to measure the sputter yield Y ; defined as the ratio of the number of ejected atoms per incident ion. The targets evaluated were molybdenum, a carbon-carbon composite, and a sample of CVD diamond. Corresponding to each value of the sputter

yield one can calculate a corresponding erosion rate which is a more meaningful number for the present comparison.

The procedure used in the experiment involved mounting the samples in a holder which is then placed in an ion beam of known energy. The samples were masked so that only a narrow area was exposed to beam. This exposed area resulted in a "trench" being eroded from the sample, the depth of which could then be measured using an Alpha-Step profilometer. The eroded depth provided a means of calculating the number of atoms removed per unit area. In addition the beam current density incident on the samples was recorded during their exposure using a biased planar probe located centrally in the sample mount. This probe was also used to map the beam current density profile in order to estimate the variation of ion flux to the various samples due to nonuniformities in the beam. A simplified schematic of the facility is given in Figure 1.

One challenge in conducting this type of erosion measurement is minimizing the influence of residual facility gases. Previous tests suggest that the presence of nitrogen can lower the erosion rate of molybdenum whereas oxygen has been shown to increase the erosion rates of carbon based materials. Reactive ion etching is one technique which has been used successfully to etch structures in diamond. This technique uses a NO_2 gas jet incident on a diamond surface under bombardment with 2 keV xenon ions. The incident ions in this case provide the activation energy required to form CO and CO₂ volatiles¹⁰. Minimizing contamination is therefore essential to reduce the chances of reactively eroding the carbon targets.

For these reasons the present test was conducted in a facility capable of relatively low ultimate pressures. The system used was an ultra-high-vacuum chamber with a 3 cm Kaufman (Commonwealth Scientific Corp.) ion source normally used for sputter deposition. The vacuum chamber geometry is depicted in Figure 1.

The system is pumped with a turbomolecular pump backed by a small mechanical pump down into the 10^{-6} Torr range and then with an 8" cryopump to the 10^{-9} to 10^{-10} Torr range. The chamber is wrapped with resistive heaters connected to an automatic timer which bakes it out every evening for several hours.

One drawback of using such a small chamber is the relative inflexibility of sample and probe placement. For these tests the samples were located approximately 2.5" downstream of the source exit plane. Although this resulted in relatively high incident current densities, and hence shorter test times, it also resulted in a less than optimal uniformity of the current profile over the area where the samples were located.

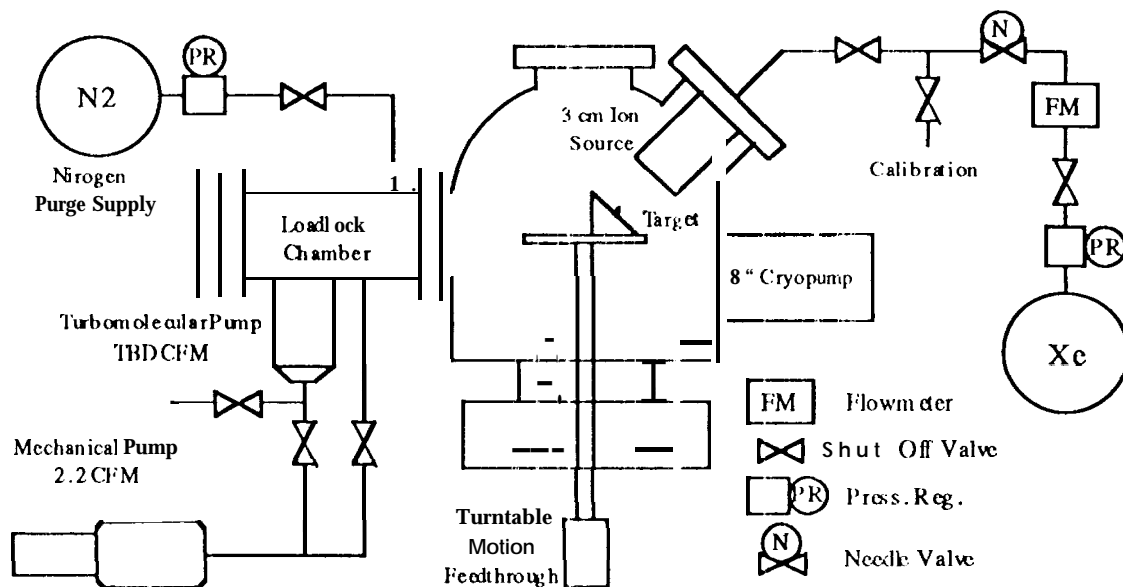


Figure 1. Schematic of the UHV Test System

Results

The results of the erosion measurements for tests conducted with beam voltages of 500 V and 750 V are presented in Table 4. For comparison, the sputter yield for molybdenum is also presented from References 3 and 4. The erosion rates listed have been normalized to an incident current density of 1 mA/cm^2 for the purposes of comparison.

The duration of the 500 V test was 5.04 hrs. and the 750 V test 1.98 hrs. The recorded probe current density as a function of time was integrated to determine the incident ion density used in calculating the sputter yields.

The carbon-carbon used is commercially available P-100 manufactured by TBD and the diamond used was a plasma assisted chemically vapor deposited (PACVD) sample obtained from Crystalline Materials Corp. (CMC). The diamond sample provided to us was undoped and had a mirror finish obtained through a process proprietary to CMC.

The data in table show a higher than predicted yield for molybdenum based on previously published data. The yield is approximately 46 percent higher than the published value at beam energy of 500 eV and 12 percent higher at an energy of 750 eV. If it is correct that contamination with nitrogen has the effect on molybdenum of lowering its erosion rate then if contamination had been present, the measured yield should have been lower, not higher than previously published values.

Another quantity in which uncertainty is introduced is the incident ion current. During the experiment, the probe is centered on the point of maximum current in the beam. This was checked at various times during the experiment by moving the probe slightly and observing the probe current. The eroded portions of the samples are located within a 0.5 inch diameter circle centered on the probe. Non uniformity of the beam results in the current density decreasing away from the probe. As a result, using the current measured by the probe for the yield calculations would tend to under estimate rather than over estimate the sputter yield and therefore would not account for the difference with previous results.

The agreement seems reasonable, particularly at the higher energy, given the lack of information available regarding the original experiments in references 3 and 4. In particular it would be useful to know the base pressures and what steps were taken to minimize the chances of contamination.

Due to differences in density, the relative erosion rates is a more, meaningful comparison than the yield for the purpose of evaluating a grid material. At 500 V the ratio of erosion of molybdenum to carbon-carbon was 7.3:1 and the ratio of carbon-carbon to diamond erosion was 1.6:1. At 750 V the ratio was 7.7:1 for molybdenum to carbon-carbon and 1.9:1 for carbon-carbon to diamond. The ratios for molybdenum to carbon-carbon are lower than one would expect from previous experiments by Meserole¹⁰ which at an accelerating voltage of 300 V was closer to 15:1. If air seepage had occurred the erosion rate of the carbon based materials would have higher as discussed

Table 4. Measured Sputter Yields and Erosion Rates for
Xc Ion Bombardment of Various Target Materials

Material	500 eV		750 eV	
	Y ^a (atoms/ion)	Erosion Rate ^{a,c} (Å/rein)	Y ^b (atoms/ion)	Erosion Rate ^{b,c} (Å/min)
Molybdenum	1.04	608	1.27	743
Molybdenum (Ref. 4)	0.71	415	1.13	661
Molybdenum ^d (Ref. 3)	0.90	526	—	—
Carbon-Carbon	0.181	83	0.218	96.2
CVD Diamond	0.243	52	0.246	52.5

^aBase Pressure = $1.1 \cdot 10^{-8}$ Torr, Ave. current density = 2.17 mA/cm^2

^bBase Pressure = $2.3 \cdot 10^{-9}$ Torr, Ave. current density = 3.43 mA/cm^2

^cNormalized to a current density of 1 mA/cm^2

^dData available only up to an energy of 600 eV

earlier although the erosion of molybdenum would probably have been lower. To minimize the chances of air contamination, a base pressure of 10^{-8} to 10^{-9} Torr established before each run and the chamber which is only vented when a sample is being exchanged, was baked out every evening.

IV. Fabrication

Diamond CVD

The deposition of diamond film from the vapor phase has been an area of significant research activity for several decades. A discussion of the gas and surface chemistry as well as the various reactor technologies used is beyond the scope of this paper. Nevertheless it is useful to outline the processes involved and identify at least one plausible deposition approach for the purpose of establishing the feasibility of using diamond for this application.

The basic elements important to the diamond deposition process are depicted in Figure 2. A gas phase source of carbon such as methane is introduced to a chamber along with a diluent gas such as hydrogen. Typically the methane is only a few percent of the total gas flow by volume. Some source of energy is needed to dissociate and activate gas species into various species essential to the growth chemistry. Many activation sources have been employed successfully including RF, microwave, hot filament, flame, and DC arc plasmas.

The substrate can be any of a variety of materials with molybdenum a common choice. Diamond crystallite will

nucleate on the substrate after a period of time which depends upon numerous variables including substrate temperature, surface preparation (such as scratching) and gas phase species concentrations. As adjacent crystallite grow together a continuous film is formed. The growth rate of the film will depend on the type of process used but can range from less than a micron per hour for a hot filament reactor to hundreds of microns per hour for DC arcjet plasma sources¹².

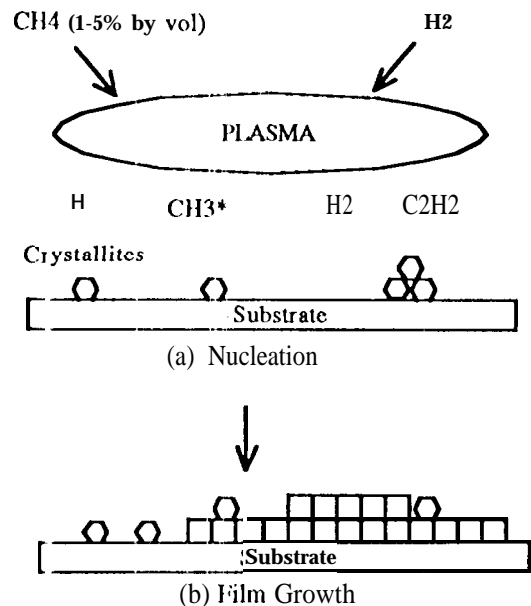
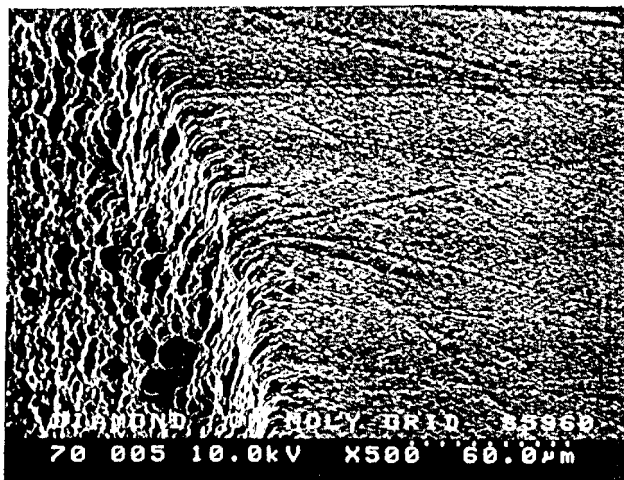
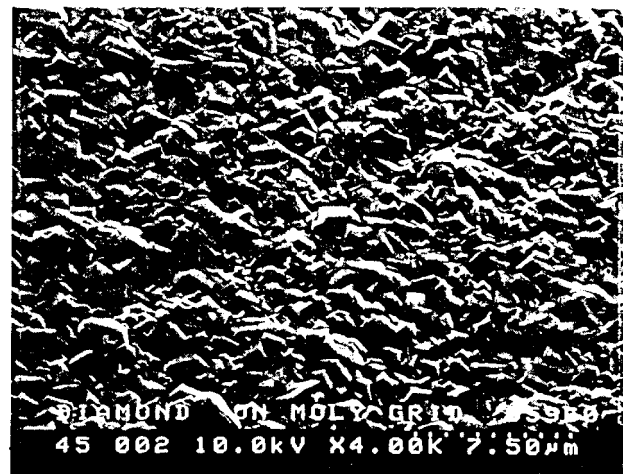


Figure 2. Plasma Enhanced Diamond Chemical Vapor Deposition



3a: Deposition in region bordering hole



3b: Deposition on surface of webbing

Figure 3. SEM Micro graph of Chemically Vapor Deposited Diamond on Molybdenum Grid with 3mm holes

For more detailed information the reader is referred to an excellent review article by Spear¹³ which discusses properties of diamond and deposition chemistry. Also the review by Bachmann and Enckevort¹² details the relative merits of the various deposition technologies.

Diamond Coated Molybdenum Grid

Molybdenum is commonly used successfully as a substrate for diamond deposition. In addition molybdenum has been used in the fabrication of ion thruster grids for many years. For these reasons deposition of diamond directly on a molybdenum grid to form a coating emerged early as a option to obtain some of the benefits of diamond.

The advantages of this scheme include the structural rigidity afforded as a result of the metal. In this case the diamond acts primarily as a sputter resistant coating which may be only a few tens of microns in thickness. A sample of molybdenum grid material from an older generation ion thruster (3 mm hole diameter) was used in one experiment to evaluate the growth of diamond film in the region along the walls within the holes and on the face of the webbing. This deposition was performed in an oxygen-acetylene flame diamond synthesis reactor at Caltech. In this reactor, the substrate sits directly below a planar flame during deposition. The gas flows downward at the substrate resulting in a stagnation flow. The flow geometry establishes a species concentration boundary layer above the substrate through which methyl and atomic hydrogen can diffuse to the growing film surface.

The conditions of the deposition are listed in Table 5 and the SEM micro graph in Figure 3a shows a region adjacent to one of the holes. The spherical features within the hole are probably non diamond carbon while the

crystalline structures on the face of the webbing and seen at higher magnification in Figure 3b are probably diamond. The presence of the non crystalline deposits in the hole suggest the lower temperatures and/or gas species concentrations unfavorable for diamond formation. The erosion due to CEX ion impingement is expected to occur in the webbing between holes and therefore the formation of diamond on this surface as evident in Figure 3b was encouraging.

Table 5. Diamond Deposition Conditions for Trial on Molybdenum Grid (Oxygen/Acetylene Flame Reactor)

O ₂ /CH ₂ Ratio	1.165
Substrate Temp	8-10 900C
Burner-Substrate Distance	10 mm
Deposition Time	3 hrs.
Surface Preparation	scratching with diamond paste

One disadvantage of coating molybdenum with diamond stems from the differing CTE. As a molybdenum substrate is allowed to cool after deposition of a diamond film, the film usually peels off as a result of the differing amounts of contraction. This could be a concern given the thermal cycling to which an ion thruster grid would be subject during a mission. However one of most important applications of diamond coatings is for machine tool bits which are subject to thermal cycling under much more severe mechanical stresses. The adhesion can be improved by depositing at substrate temperatures favorable to the

formation of intervening layers between the diamond and metal. Molybdenum carbide is one example¹⁴.

Free Standing Diamond Grid

A second fabrication option is to deposit a thick film which is self supporting. Such a free-standing diamond grid would have some important advantages over the diamond coated molybdenum.

One advantage is that the adhesion is not an issue. A second advantage is that the CTE for the grid is lower since it is not restricted to that of its metal backing. A significant drawback of the free standing grid is the loss of flexural rigidity obtained from the molybdenum. Polycrystalline diamond is exceedingly hard but also brittle. Thus far no information has been found quantifying the flexural rigidity of free standing CVD diamond films but small samples, even those approximately a millimeter thick are fragile and require careful handling.

The process for growing a free-standing grid would essentially be the same as the coated molybdenum except the film grown would have to be hundreds or thousands of microns thick and poor adhesion would be desirable in order to remove the diamond from its substrate. It may still be possible to grow the diamond on a substrate with holes in order to produce a grid with holes. However, because the thickness involved, the diamond film may grow laterally as well as vertically, thereby closing off the holes. If this is the case it would then be necessary to first produce a free-standing diamond disk, and then machine the holes, probably with an excimer laser.

An alternative to deposition on a molybdenum substrate (with or without holes) is to deposit the diamond film on a silicon substrate which has been patterned with silicon dioxide using standard lithographic techniques. The benefit here is that the film will be selectively deposited on the silicon and not on the oxide. In this way it is possible to pattern the wafer with oxide "dots" where the grid holes are supposed to be. Because the diamond nucleation density on SiO₂ is several orders of magnitude lower than on Si, the oxide essentially inhibits the growth. Once the film has been deposited, the substrate can be removed with an acid. While this approach involves more process steps, it may be desirable if the separation of the diamond from a molybdenum substrate with holes is difficult to accomplish without damaging the webbing between the holes.

Diamond Coated Carbon-Carbon Grid

One last promising option which has only received a limited amount of attention is that of coating a carbon-carbon grid with diamond film. This scheme has the advantage of potentially incorporating many of the

benefits of carbon-carbon and diamond. In particular, the carbon-carbon panel provides the flexural rigidity as in the coated molybdenum case but does not suffer from the relatively high CTE.

Deposition of diamond has been demonstrated on carbon fiber composites with the crevices between adjacent fibers providing favorable nucleation sites.

V. Conclusions

Chemically vapor deposited diamond film was evaluated as a potential material for use in ion optics. It has a combination of properties which are ideally suited for this application including a high thermal conductivity, low CTE, and low erosion rate relative to carbon-carbon composites and molybdenum. Three fabrication options were discussed; diamond coated molybdenum, free-standing diamond, and diamond coated carbon-carbon.

Coating of carbon-carbon is promising but given the negative CTE of carbon-carbon will require evaluation to assess the adhesion of the film under thermal cycling. Deposition of free-standing films is done routinely in industry as is laser drilling of holes. The rigidity of the diamond film however could be a problem requiring special handling. Depositing a free-standing film with holes in one process step is possible using either a substrate with holes or selective deposition but will require further testing to assess the extent of growth into the holes. Coating of molybdenum grids should be feasible using existing technology and the adhesion should not present a serious problem if an intervening layer is formed.

In each of these options, p-type doping of the diamond film can be performed using diborane with the appropriate equipment. The conductivity should be sufficient for the relatively small currents the accelerator grid is expected to handle.

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